

PROJECT NUMBER : 2520
PROJECT TITLE : Flavor Research
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I. FLAVOR RELEASE TECHNOLOGY

A. Objective: To investigate the synthesis and pyrolysis of various flavor release systems for use in new or improved products.

B. Results: We continue to explore the grafting of the trans-2,3-cyclic carbonates of glucose derivatives to other carbohydrates using relatively simple molecules as model compounds. Methyl 4,6-O-benzylidene- α -D-glucopyranoside 2,3-O-cyclic carbonate was prepared from methyl 4,6-O-benzylidene- α -D-glucopyranoside and triphosgene in 60% isolated yield. Reaction of methyl 4,6-O-benzylidene- α -D-glucopyranoside 2,3-O-cyclic carbonate and n-butanol was carried out in chloroform and pyridine. In both solvents, the expected two isomeric carbonates, methyl 4,6-O-benzylidene-2-O-butoxycarbonyl- α -D-glucopyranoside and methyl 4,6-O-benzylidene-3-O-butoxycarbonyl- α -D-glucopyranoside were obtained in about equal amounts. However, the rate of reaction was much slower in pyridine than in chloroform.

Reaction of methyl 4,6-O-benzylidene- α -D-glucopyranoside 2,3-O-cyclic carbonate and phenethyl β -D-glucopyranoside was also carried out in chloroform and pyridine. In both cases, a complicated reaction mixture was obtained. The reaction in chloroform was analyzed and two components were readily identified as phenethyl β -D-glucopyranoside and methyl 4,6-O-benzylidene- α -D-glucopyranoside, the decomposition product from methyl 4,6-O-benzylidene- α -D-glucopyranoside 2,3-O-cyclic carbonate. Two other products were also isolated and identified, namely methyl 4,6-O-benzylidene-2-O-(phenethyl β -D-glucopyranoside 6-O-carbonyl)- α -D-glucopyranoside and methyl 4,6-O-benzylidene-3-O-(phenethyl β -D-glucopyranoside 6-O-carbonyl)- α -D-glucopyranoside in about equal amounts. Other products present in the reaction mixture have not yet been isolated. The reaction in pyridine is much slower and some decomposition of methyl 4,6-O-benzylidene- α -D-glucopyranoside 2,3-O-cyclic carbonate to methyl 4,6-O-benzylidene- α -D-glucopyranoside is also observed. Analysis of other components is in progress.

We have received an ultrasound generator and its associated accessories and reactors. The instrument has been checked out and preliminary investigation is being carried out.

We continue to explore the synthesis and pyrolysis of various menthol release agents. The magnesium and calcium salts of (+)-di-O-acetyl-L-tartaric acid mono-menthyl ester have been synthesized and submitted for TGA. The mono menthyl ester of glutaric acid has been prepared and submitted for NMR. A large scale preparation of mono menthyl itaconate has been carried out to make 440 grams of product. The method of synthesis was improved to give an 85% yield vs. previous yields which averaged about 60%. A set of trials were run with G. Keritsis on the use of mono menthyl itaconate as an encapsulating agent for menthol. The results of these trials

2022201724

were very promising. Menthol was encapsulated by cocrystallization. It was shown that these crystals can be hardened and stabilized by surface formation of the calcium salt of the itaconic acid. A series of experiments have been planned to develop this concept and provide materials for evaluation.

A new class of menthol release compounds derived from beta-hydroxy carbonyl compounds is being developed. A first example has been prepared and characterized (CR-2956). This is the menthyl carbonate of methyl 3-hydroxy-3-phenyl propanoate. Pyrolysis GC/MS at 300° shows clean decomposition to menthol and methyl cinnamate.

The preparation of a flavor-release agent for 3-vinylpyridine is being pursued. The latter is a marker for gas phase sidestream smoke. Reduction of 3-acetylpyridine gave the expected alcohol. Reaction of the alcohol with oxalyl chloride gave the oxalate ester. Preliminary results indicate thermal decomposition by a radical process giving 3-ethylpyridine and cyclobuta [b]pyridine instead of the expected 3-vinylpyridine. The analogous menthyl carbonate is in preparation.

II. FILTER MATERIALS AND PAPER TECHNOLOGY SUPPORT

- A. Objective: To find outside suppliers for large quantities of new filter materials and inorganic paper additives.
- B. Results: Hydrothermal production of magnesite at R.S.A. has been suspended. Recent results have indicated that Hasteloy B-2, a high nickel alloy, may be being corroded during the synthesis, resulting in unacceptably high levels of nickel in the final product. While it was thought that R.S.A.'s large pressure reactor was manufactured solely from 316L Stainless Steel, on careful checking, it was determined that the reactor had Hasteloy B-2 baffles welded to the walls. Until it has been conclusively shown that the process does not attack Hasteloy B-2, no further work will be scheduled at R.S.A.

Four major and two intermediate suppliers of bulk quantities of the inorganic compounds required for production of eitelite were contacted. Samples and the appropriate technical information on their materials was requested and has begun to arrive.

A sample of partially acetylated wood pulp and surface deacetylated two have been received from Courtaulds. The material look good and handsheets will be made in the coming weeks.

2022201725